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Materials Research Department annual report 2003

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Publication date:
2004

Document Version
Publisher's PDF, also known as Version of record

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Materials Research Department
Annual Report 2003

Risø National Laboratory

April 2004

Risø-R-1451 (EN)

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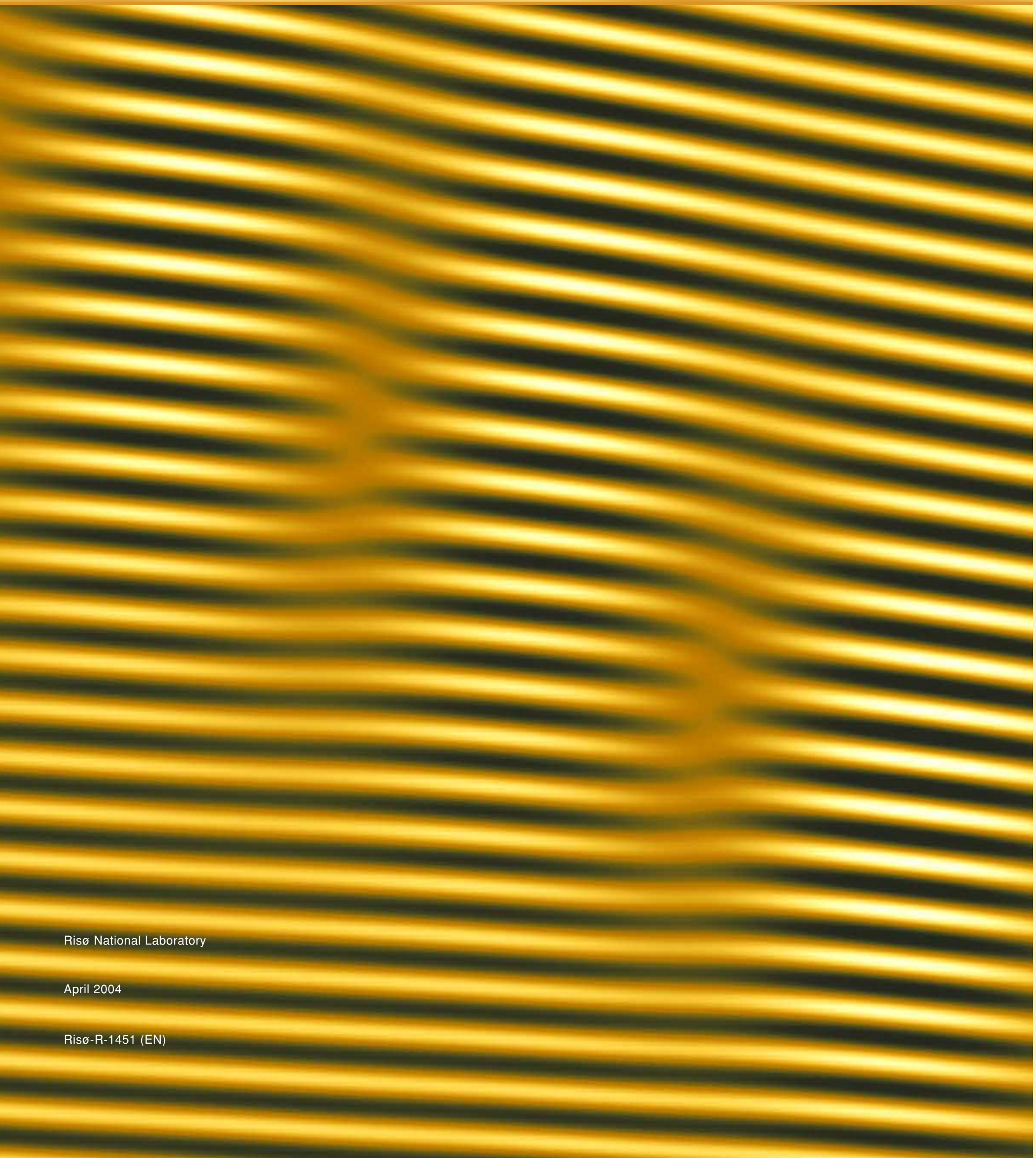

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c o n t e n t s

abstract

Selected activities of the Materials Research Department at Risø National Laboratory during 2003 are described. The Scientific work is described in five chapters and a survey is given of the Department's educational activities along with a list of published work, prizes, organized meetings, and membership of committees. Furthermore, the main figures outlining the funding and expenditures of the Department are given and a list of staff members is included.

Risø-R-1451 (EN)

Published by the Materials Research Department,
Risø National Laboratory, April 2004.

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Photos:	Michael Daugaard Boye Koch Preben B. Olesen Katrine Nørgaard Toft a.o.
Layout:	Pernille Dam · pdsign.dk
Printing:	Center-Tryk A/S
Front cover:	Fourier filtered HREM lattice image obtained in a JEOL 3000F electron microscope showing individual dislocations in a grain boundary in a nickel sample heavily deformed by high pressure torsion. Magnification ~ x 50,000,000
Inside cover:	Autumn view from meeting room 1.

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ISBN 87-550-3299-0
ISBN 87-550-3300-8 (internet)
ISSN 0106-2840
ISSN 1397-8071

2003 - New Directions

The role of science in society is undergoing significant changes. Science is expected not only to create new knowledge, but also to take responsibility for turning this knowledge into technology and innovation, to create new wealth and new jobs. Risø National Laboratory is committed to this new role in the Danish society and has put high priority on education and innovation in 2003. Our new target is not only 'impact on science', but also 'impact on society'. In the Materials Research Department we will take the responsibility to assure that the science we perform will be used in society. This can only be achieved by having close interactions with industry and innovators, for example as is the case with our activities within solid oxide fuel cells. In parallel with our development of new materials for solid oxide fuel cells, we are scaling up the capability for mass production of cells, in close collaboration with the company Haldor Topsøe A/S. In 2003 we increased our production capacity of solid oxide fuel cells dramatically. This was a very important milestone for us and for our industrial partner.

The role and mission of the Materials Research Department has become wider and we have put more emphasis on education and innovation in 2003 and will continue to do so. Young students are keen to learn and to understand how science – in particular their field - can be used in society. We have had many good and motivated students attending our courses in fuels cells, materials, metals and superconductivity. A new approach has been taken in 2003 by offering projects to students with a more business-oriented background in order to combine science and business into innovation.

The major research objective of the Materials Research Department is the development of advanced materials for new energy technologies, in particular fuel cell and hydrogen technologies; as well as investigation and development of new materials related to e.g. superconductivity, and light and strong materials in demand within e.g. wind energy technology. An integral part of our research is the use of large-scale facilities for X-ray synchrotron radiation and neutron scattering. Furthermore, the Departments serves as a national centre for electron microscopy, a role that will be further strengthened with a new field emission scanning electron microscope granted by the Villum Kann Rasmussen foundation.

Leaders ready to move.

People on the floor.

We have also taken up new directions in 2003 outside the main focus on energy and used our competence and know-how in a number of smaller projects in particular within the health and safety sector. In the medical device area we have seen that our background in advanced materials gives a perfect match to the need for invention of new devices and implants. We will put more focus within this area in the coming years, in particular because the potential for innovation and spin-off companies is very high.

At the same time, it is important also to keep our core skills at the international forefront of science, by publishing in peer reviewed journals. Traditionally, one of our expertises is developments of new experimental and analytical tools. One of the highlights has been the development of the 3D X-ray microscope, which now has a spatial resolution of a few microns inside a material. This equipment is located at the European Synchrotron Radiation Facility, Grenoble, France. For the first time it has been possible to record the growth of a single grain inside a metal and discover how irregular the growth is. Furthermore, the new transmission electron microscope that was inaugurated in 2002 has been crucial for a number of projects within nanoscience and nanomagnetism.

In collaboration with the Danish Polymer Centre we were awarded a framework program from the Danish Technical Research Council with the title 'Interface Design of Composite Materials'. This will enable us to go from the more macroscopic view of composite materials to working at the interface between fibre and matrix on the nanoscale. Furthermore, an application for a pulsed laser deposition equipment for ceramic oxides was approved and partly funded by the Danish Technical Research Council.

Superconductivity and magnetism was the theme of the 24th International Risø Symposium on Materials Science. We were very pleased that we were able to attract some of the world's most prominent scientists within the field. A number of excellent reviews and research highlights were given, and the symposium was attended by 100 participants.

It is a pleasure for the Department to note that Mogens Mogensen was appointed Research Professor in Materials Chemistry with special tasks in research and development in solid oxide fuel cells, Per-Anker Lindgård was appointed Adjunct Professor in biological and nanoscale physics at the Technical University of Denmark, Des McMorrow was awarded the Allan Mackintosh Award and Martin Søgaard was awarded the 'Candidate prize from Elektrofondet' for his outstanding Master's thesis on ion conducting ceramics.

Robert Feidenhans'l

Staff
The Department comprises about 160 people, including Ph.D. students and Post Docs. Out of these, 67 are permanent scientific staff, including two research specialists and five research or adjunct professors. Two prominent staff members left the Department in 2003. Des McMorrow is as of January 1st, 2004 professor in the Physics Department, University College London. Kurt N. Clausen started January 1st, 2004 as director of Condensed Matter Research with Neutrons and Muons at the Paul Scherrer Institute in Switzerland.

Education
The Department is involved in a variety of educational activities. The Department had 27 Ph.D. students, of whom six completed their degrees in 2003, and 32 Master's and Bachelor's students. Furthermore, 34 summer students were working in the Department.

Publications
The publication list of the Department comprises 102 papers in international journals, including several in the most prestigious journals. In addition, many conference papers, books and reports were published.

Economy
The turnover of the Department is 118 Mkr. (15.8 M€) out of which 50 Mkr. (6.8 M€) comes directly from Risø, 42 Mkr. (5.5 M€) from programme supported research and 26 Mkr. (3.5 M€) from companies and commercial income.

Structure
At the end of 2003 it was decided to merge the programme for Superconductivity and Magnetism into the programme Nano- and Microstructures in Materials.
Hence, as of January 1st, 2004 the Department is organized into four programmes:

Metal Structures in 4 Dimensions
headed by Dorte Juul Jensen

Composites and Materials Mechanics
headed by Povl Brøndsted

Nano- and Microstructures in Materials
headed by Allan Schrøder Pedersen

Fuel Cells and Materials Chemistry
headed by Søren Linderoth

Sponsors
We thank all our collaborators and sponsors for the support we have received in 2003, in particular: Danish Energy Authority, Elkraft System, Eltra, Public Service Obligation, Haldor Topsøe A/S, the Danish Natural Science Research Council, the Danish Technical Research Council, the Danish National Research Foundation, Euklid, and the European Commission.

The hollow cathode sputtering system for production of single crystalline metallic nanoparticles (clusters) in the size range of 5nm to 50nm.

Superconductivity and Magnetism

Superconducting and magnetic materials are serious candidates for applications ranging from electric power devices to high-tech electronics, and they continue to attract strong attention within basic science. Although considered mutually exclusive the superconducting and magnetic phases are highly interrelated, as exemplified by the high-temperature superconductors that are magnetic insulators unless suitably doped.

The programme is centred around two framework programmes on superconductors and magnetic nanoparticles sponsored by the Technical Science Research Council, and supported by two national centres on neutron and synchrotron X-ray scattering funded by the Natural Science Research Council.

Materials development is focused jointly within basic and strategic studies. The unknown mechanism underlying high-temperature superconductivity and the prospect of a room temperature superconductor are significant incentives along with the fascinating properties of new magnetic materials, e.g. for modern computer technology. The scientific challenges and the broad range of applications were clearly outlined during the 24th Risø International Symposium on Materials Science dealing with *Superconductivity and Magnetism: Materials Properties and Developments.*

In the materials development programme the influence of chemical substitutions on the superconducting properties of MgB_2 has been studied, and promising materials have been processed to high-quality steel-clad tapes. New materials prepared and studied include the so-called spin-ladder compound $(Sr,Ca)_{14}Cu_{24}O_{41}$ doped with Bi and Sc. They resemble high-temperature superconductors but finite ladders and networks replace the infinite copper-oxide network. Basically they are magnetic insulators but superconductivity has been reported in literature. In the search for new types of nanomagnetic materials a highly unusual onion-shell structure has been observed in nanoparticles produced by the micellar method.

Synchrotron X-ray diffraction has been performed at HASYLAB in Hamburg using high-energy photons that allow for in-situ characterization of texture and phase transformations during thermal processing of MgB_2 tapes in steel-clad and $(Bi,Pb)_2Sr_2Ca_2Cu_3O_{10}$ in silver-clad. This technique has also been used for basic studies of the charge distributions in $YBa_2Cu_3O_{6+x}$ and $La_{2-x}Sr_xCuO_4$ type superconductors.

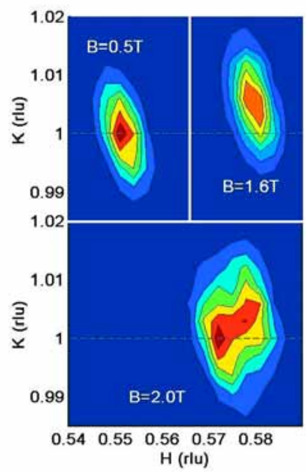
Neutron scattering is used extensively for basic studies of the static and dynamic magnetic properties. The neutron spectrometers, which were transferred from the closed DR3 reactor to the Paul Scherrer Institute in Switzerland, have been upgraded to fulfil the collaboration contract and assure access to neutron scattering facilities at a high international level. The neutron scattering studies include determination of magnetic phases in superconducting RNi$_2$B$_2$C (R = Er and Tm) and the magnetic properties of the vortex structure in $La_{2-x}Sr_xCuO_4$. By neutron scattering several new nano-scale effects have been observed in magnetic nanoparticles; for instance it has been shown that in isolated hematite nanoparticles the superparamagnetic relaxation rate saturates abruptly by increasing temperature, and the discreteness of the spin waves has been observed.

Niels Hessel Andersen

Magnetic structure in a superconducting environment

Although superconductivity and magnetism are considered mutually exclusive states they are found to co-exist in some materials. An interesting example is $ErNi_2B_2C$ which becomes superconducting below T_c =11 K and orders antiferromagnetically at T_N = 6 K. We have studied the magnetic structures of $ErNi_2B_2C$ with neutron diffraction while superconductivity is suppressed by applying a magnetic field. The magnetic structure contains ferromagnetic sheets of Er-moments aligned along the [0 1 0] direction of the tetragonal crystal structure and antiferromagnetically modulated along [1 0 0]. In zero and low field the modulation vector is [H 0 0] where H = 0.55 in reciprocal lattice units (rlu). The figure shows neutron diffraction contours measured at 1.8 K with the field applied along [1 1 0]. The low field phase with H = 0.55 is presented in the upper left panel. The upper right part presents the diffraction pattern at 1.6 T. Here the period is H = 0.58 rlu, and remarkably the structure is rotated away from the symmetry direction by 0.5 degrees. The diffraction pattern for the 2.0 T case is shown in the lower panel. Two different ordering vectors can be observed, one with H = 0.57 rlu, and the other one with H = 0.58 rlu. Notice, that only one of the reflections is rotated. We understand many of the observed features based on a theoretical model that shows stability of long-period commensurate structures by formation of phase slips in the simple antiferromagnetic ordering corresponding to H = 0.5. The transverse component of the ordering vector may possibly be explained by a rotation of the magnetic moments that lowers the Zeeman energy. So far we have not observed any clear influence hereof on the superconducting properties.

Katrine Nørgaard Toft

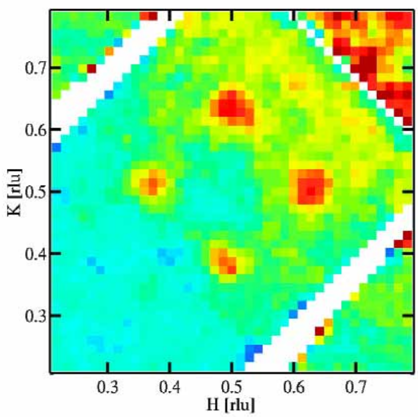


Neutron diffraction patterns of magnetic structures in $ErNi_2B_2C$ at 1.8 K with the magnetic field applied along [1 1 0]. The modulation vectors [H 0 0] where H = 0.55 rlu for B=0.5 T, H = 0.58 rlu for B=1.6 T, and H = 0.57 and 0.58 rlu for 2.0 T. Notice that the modulation vector is rotated 0.5 degrees away from the symmetry direction for the 1.6 T and partly for the 2.0 T data.

Imaging spin fluctuations in a high-temperature superconductor

Neutron scattering studies of the magnetic properties of high-temperature superconductors and in particular those of $La_{2-x}Sr_x CuO_4$ have been pivotal in attempts to understand the novel physics governing the exotic behaviour of these materials. The technique takes advantage of the dipole-dipole interaction between the neutron spin and the magnetic moments residing on Cu atoms. The figure shows a colour map of the magnetic correlation in $La_{1.84}Sr_{0.16}CuO_4$ at an energy of 10 meV for a temperature of 10K. The data were obtained using the MAPS time-of-flight spectrometer located at the ISIS spallation source in the UK, and the samples studied were grown in an optical image furnace as the one owned by the department. In the image shown, the four-fold symmetry around the antiferromagnetic $\mathbf{q}=(1/2,1/2)$ point reflects the existence in these materials of a so-called stripe phase. This consists of periodically spaced, metallic rivers of charge separated by insulating regions with local antiferromagnetic order. Because the crystals are twinned, there are four rather than two peaks. A leading class of theories claim stripes to be a central ingredient in the mechanism underlying high-temperature superconductivity. To test such ideas, it is of crucial importance to map out in detail the magnetic susceptibility $\chi''(\mathbf{q},w)$ as a function of momentum and energy transfer, and neutron scattering does precisely this. Analysis of our data has revealed dramatic changes in $\chi''(\mathbf{q},w)$ upon cooling below the transition temperature T_c=39K to the superconducting state, as well as evidence for a crossover as a function of energy away from the four-fold symmetric pattern seen in the figure. Both these observations put strong constraints on theoretical efforts to understand the high T_c conundrum.

Niels B. Christensen and Des F. McMorrow



A colour map of the magnetic correlations in $La_{1.84}Sr_{0.16}CuO_4$ at an energy of 10 meV for a temperature of 10K.

Palle Nielsen is making the final checks before a metal sheet is deformed in the rolling mill. Subsequently, the influence of the deformation process on the microstructure is characterized carefully using advanced characterization techniques such as SEM, TEM and 3D X-ray diffraction microscopy.

Metal Structures in Four Dimensions

Metals are among the most frequently applied materials and are used on all scales from large constructions such as bridges to nanoscale devices such as miniature gears, diagnostic tools and implants. Improved and optimized metals and metal processing contribute to security, economic stability and environmental progress in our daily lives, although the impacts are not fully appreciated by those affected.

In the programme we aim at dealing in a meaningful way with real heterogeneous metal structures rather than imposing homogeneous approximations. This aim is approached by combining advanced experimental characterizations with physically based modelling.

Key experimental techniques include electron microscopy and 3 dimensional X-ray diffraction (3DXRD) microscopy by high energy X-rays from synchrotron sources. These two types of experimental techniques are used generally in combination to achieve the best possible characterization of a given scientific problem, and synergy is often obtained.

Concerning the experimental techniques, positive news in 2003 were:

A very positive international evaluation of the beamline ID11 at ESRF, Grenoble, France, where the 3DXRD is situated. The evaluation emphasised the importance and potentials of the 3DXRD.

The Villum Kann Rasmussen Foundation decided to support an application for a modern field emission gun scanning electron microscope (FEG SEM) equipped with electron backscatter pattern (EBSP) and energy dispersive spectroscopy (EDS) facilities.

During the year, good scientific progress was achieved on all scientific fronts. Examples of highlights are:

The dynamics of structural units as small as 200nm can be followed in-situ by 3DXRD.

The flow stress, calculated from microstructural parameters by assuming that stress contributions for dislocation boundaries and high angle boundaries are linearly additive, agrees well with experimental values for both deformed and annealed aluminium with structural scales from a few hundreds of nanometers to tens of micrometers.

For the first time ever the growth of a bulk nucleus has been seen in-situ during recrystallization by 3DXRD mapping.

Based on 3DXRD and electron microscopy results, it has been shown that the initial crystallographic orientation of a grain determines its rotational behaviour during plastic deformation.

In spite the fact that the Programme focuses on metals, the outcome may well stretch beyond metals, and the methods developed may be used for other material classes as well. As an example of the latter, 3DXRD has been used for studies of materials as different as ceramics and ice by external users. A prominent example was the start of a talent project, supported by STVF, aimed at studying fracture and fibre debonding in polymers. Here use will be made of a marker tomography method established for determination of strain in metals; described in one of the following highlights.

Dorte Juul Jensen

3D strain in fibre reinforced materials

A novel method to allow strain measurements within bulk material has been developed. The method is based on micro-tomography and samples that contain embedded marker particles having an atomic number significantly different than that of the matrix material.

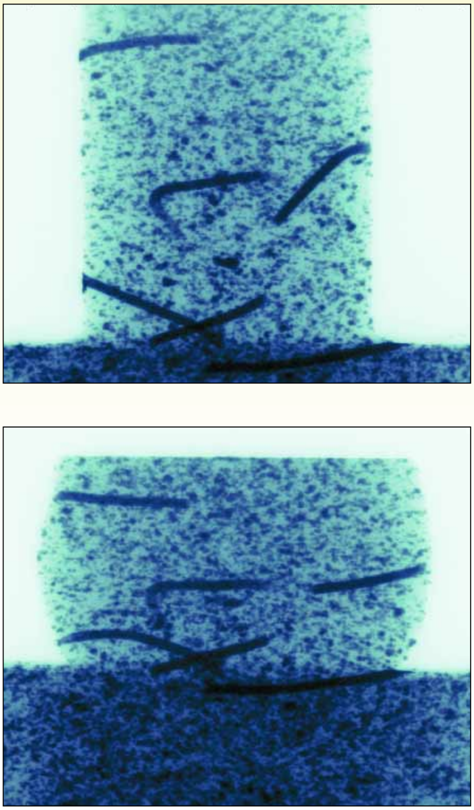
The lower limit for the size of the marker particles is determined by the spatial resolution of the X-ray detector. At present, the best X-ray detectors for tomography experiments can detect particles of a diameter exceeding 0.7 µm.

By deforming a sample that contains marker particles and identifying each particle both before and after the deformation the displacement field of all the particles can be determined. This requires a procedure for automatic identification of the particles, which is a non-trivial task due to the large number of particles.

The method for determining strain in 3D is universal in the sense that it can be applied on all materials that contain marker particles with an X-ray absorption coefficient different than the matrix. The material under investigation can be crystalline as well as amorphous.

The Center for Fundamental Research: *Metal Structures in Four Dimensions* is applying the technique to investigate the local strain within individual grains in a deforming polycrystal. In addition a new STVF funded project was initiated in 2003 with the aim of studying strain fields in fibre reinforced materials. The mechanical properties of fibre composites depend strongly on the mechanical properties of the fibre/matrix interface. By determing the strain field in three dimensions around individual fibres in a fibre reinforced composite it will be possible to gain new information about how fibres interact with the matrix material. In case of fracture new information will also be gained on fracture mechanisms, and this project will therefore contribute to the development of new and better fibre reinforced materials.

Søren Fæster Nielsen



X-ray absorption images of a cylindrical aluminium sample at 0% (top) and 40% (bottom) compression. The sample contains fibres and marker particles of tungsten.

Direct observation of subgrain evolution during recovery

During plastic deformation of metals, metastable dislocation structures form with dislocation boundaries separating nearly dislocation free regions, referred to as subgrains. The processes occurring during subsequent annealing, such as the coarsening of the microstructure, are commonly summarized as (static) recovery.

A method that enables studies of the individual *embedded* subgrains during recovery has been developed. The method is an extension of the recently developed 3-dimensional X-ray diffraction (3DXRD) method, which has been used for structural characterization of grains within millimetre-to-centimetre thick polycrystals.

A study of an *in-situ* recovery experiment on a disc of 38% cold-rolled aluminium has provided growth curves for 9 cells during annealing at 300ºC. Six of the nine growth curves are presented in the figure. The study partly proves the feasibility, partly provides the first results on the microstructural dynamics.

Among the subgrains, more than half exhibit essentially no growth, two shrink during the first hour and then stay constant, one grows rapidly during the first 5 minutes then stagnates, while the last one first grows, then shrinks. Strikingly, there is no obvious correlation between subgrain volume and growth behaviour. The smallest subgrain grows substantially during the first 5 minutes. So does the rather large subgrain. On the other hand, one of the larger subgrains shrinks during the observation and the largest one does not change in size at all. This behaviour is in marked contrast with elementary theories of curvature (i.e. interfacial energy) driven coarsening, where subgrains larger than average are expected to grow while subgrains smaller then average are expected to shrink. However, from the small number of observed subgrains it cannot be excluded, that a curvature driven model is valid in a statistical sense. Analysis of a larger number of subgrains is currently in progress aiming at resolving this issue.

Carsten Gundlach

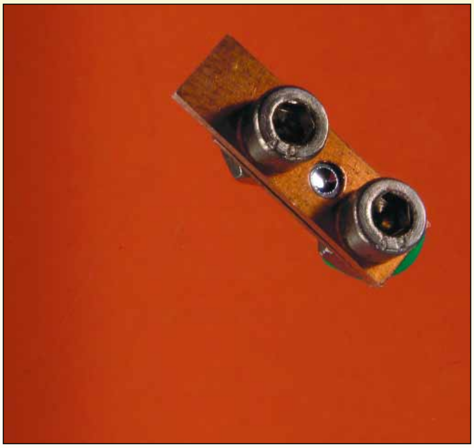
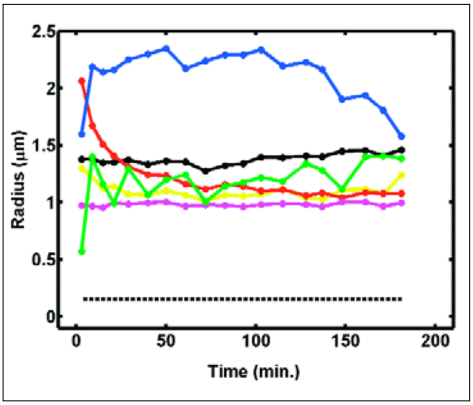
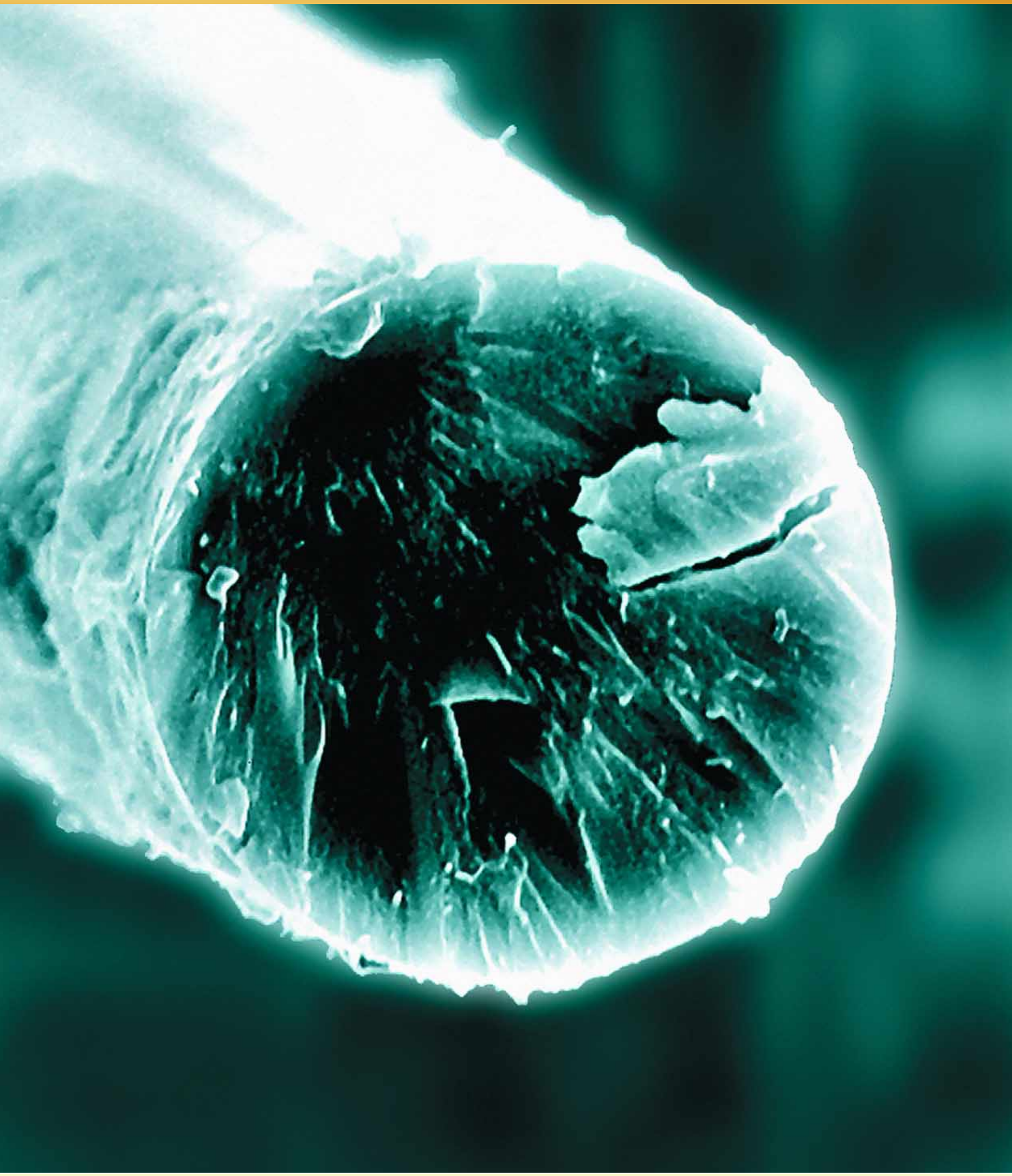


Photo of the sample holder containing the ~80 µm thick aluminium foil (diameter 3 mm) investigated with the 3DXRD microscope. Gold markers deposited next to the sample foil (not visible in this photo) are used to ensure a positional accuracy better than 1 µm. This is essential for the study of the kinetics of the individual ~1 µm sized subgrains.



Subgrain radius as a function of annealing time at a temperature of 300ºC. The dotted line represents the detection limit of the instrument.



Glass fracture pattern as observed after a single-fibre test in the SEM (15 kV). The crack initiates from a flaw origin and leads to a typical fracture pattern as the crack front accelerates. The diameter of the fibre is 14 µm. The delicate work was undertaken by A. Thraner during a summer student project.

Composites and Materials Mechanics

New techniques for destructive and non-destructive testing are introduced continuously within the Programme and brought to application in quality control and acceptance testing for external partners. Non-destructive testing of cracking along the skin/core interface in sandwich specimens under general edge loads was analysed. Numerical modelling of damage evolution in laminated composite materials has been initiated. Advanced fracture mechanics testing and analysis of mixed mode crack propagation were developed. In addition to the highlights described separately on the next pages on work with natural fibre composites and on damage mechanics, key results of the year include the following.

The tensile strengths of glass fibre reinforced composites containing sized and unsized *single* glass fibres have been examined. Fracture mechanics were applied to estimate the original flaw size and relate the observed fracture mirror surfaces to the fibre strength. Based on the observation of surface flaws, a "healing" mechanism by the sizing is considered likely.

Static mechanical properties and fatigue properties have been measured on reference wind turbine blade glass/epoxy material. This investigation is a part of the EU 5th frame programme project OPTIMAT-blades in collaboration with a consortium of laboratories and industries in Europe. The effect of extreme conditions such as temperature and humidity are investigated. The test results give the basic data for a continuum damage mechanics analysis.

A 15 million cycles (≈ 15 years) wear test of a hip implant material has been performed. The test was carried out in Bovine serum following a simplified walk loading sequence with combined torsion movement and axial loading. Tests were run at a frequency of 2 Hz, and the change in thickness of the material in the worn area was measured. Based on geometrical measurements it is estimated that less than 300 mg of material has been worn off during the 15 years wear test of the implant.

Acoustic emission has been used for checking wind turbine blades during loading for evidence of damage development. The audio feedback from the sensors and the clear visual displays (lab-view based programming) showing where damage is developing in the blade, gives the operator an indication of how the test is proceeding. The system was developed in the frame of SOCRATES WEAO programme called "Structural Health Monitoring Systems for Military Platforms – Requirements, Design, and Demonstrations (AHMOS)".

A EUCLID-project on "Survivability, Durability and Performance of Naval Composite Structures" with participation from six European nations has been completed. The project has focused on a superstructure for a Frigate type naval vessel, and all results and achievements have been compiled in a comprehensive *Guideline* covering verification and structural reliability, materials modelling and characterisation, manufacturing, structural design, fire performance and electromagnetic shielding.

Processing technologies of products for Danish industrial partners were developed successfully. In cases, which involve commercial research and development projects of innovative character a strict confidentiality is kept between the contractors.

The programme has further been active in the educational sector. A Ph.D. student working on structural properties and crack initiation in ultra high strength tool steels graduated, and graduate students (B.Sc. and M.Sc.) made their theses under supervision from members of the Programme. And at the Engineering College of Odense a new course entitled "Composite materials and processing" was given. It was directed towards students studying for B.Eng. in Integrated Design.

Povl Brøndsted

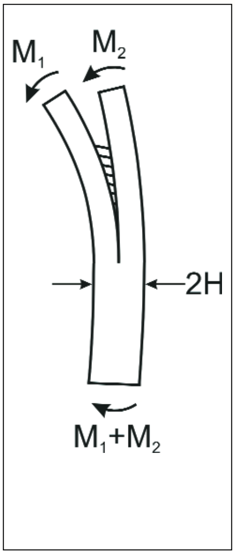
A new method for mixed mode fracture mechanics testing

A new method for fracture mechanics characterization was developed. It is a Double Cantilever Beam specimen loaded with Uneven Bending Moments (DCB-UBM). The method allows stable crack growth and, thus, is suitable for characterization of brittle materials, interfaces as well as materials having rising crack growth resistance (R-curve behaviour). Both the energy release rate and mode mixity (a measure of the relative amount of tangential and normal crack opening displacement) are independent of crack length. This is advantageous as it leads to stable crack growth under constant conditions, consequently many data points can be obtained from a single experiment.

The mode mixity is varied, simply, by altering the ratio between the two applied moments. The specimen is also suitable for characterizing large-scale crack bridging; energy release rate (the *J* integral evaluated along the external boundaries) was obtained in closed analytical form, expressed by the applied moments.

A special test set-up, based on wires and rollers, was developed for applying uneven bending moments to the DCB-specimens. A laminate/adhesive joint system was characterized by the new test method. The measured fracture resistance was considered a material property. The fracture resistance was used for predicting the load-carrying capacity of structures significantly larger than that of the test specimens. Independent measurements were made to establish the load-carrying capacity of medium size (~ 2 meters in length) adhesive joints. The measured strength values were found to be in very good agreement with the predictions.

Bent F. Sørensen



Left: Jens Olsson demonstrates the DCB-UBM test: Mixed mode cracking in a specimen consisting of glass-polyester laminates bonded by an adhesive. Right: Loadings and geometry of the double cantilever beam specimen loaded with uneven bending moments (DCB-UBM), M_1 and M_2. The thickness of the specimen is 2H.

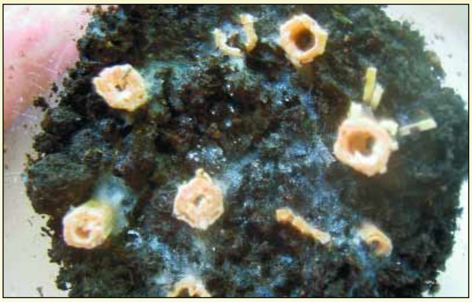
Hemp fibres for composites: crystallinity and purification

Composites are made with hemp fibres, which have high tensile strength (500-1000 MPa), low density and more sustainability than glass fibres. The fibre orientation is maintained during the processing to obtain unidirectional composites. X-ray diffraction is used as characterization method and the fibres are purified using a fungus. The X-ray diffraction was done in co-operation with K. Ståhl and J. Oddershede at DTU and the fungal treatment in co-operation with G. Daniel at WURC in Sweden.

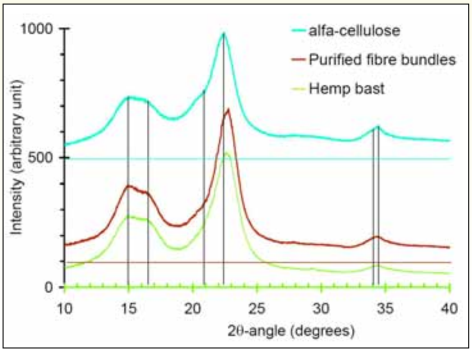
The applied hemp bast consists of fibre bundles surrounded by pectin rich cells. This hemp bast contains 64% cellulose, which is a partly crystalline polymer due to hydrogen bonds between the individual molecules. The crystalline fraction is estimated by X-ray diffraction. Amorphous cellulose, hemicellulose and lignin are diffracting, mainly, in a band of 2θ-angles between 14º and 22º. The fraction of crystalline cellulose was calculated from a linear combination of the calculated diffractogramme for crystalline cellulose and the diffractogramme for amorphous lignin fitted to the minimum intensities between the peaks. The fraction of crystalline cellulose in the hemp bast and the purified fibre bundles were 47% and 54%, respectively. Taking the cellulose fraction into account the degree of crystallinity was 73% in the hemp bast, 70% in the purified fibre bundles and 71% in the α-cellulose (a commercial cellulose).

The applied fungus was a mutated white rot fungus, which degrades the weak material in the hemp bast without cellulose degradation. The initial experiments were made with hemp stems plugged into soil as shown in the photo. Cultivation in soil is labour intensive so the procedure was later performed in liquid, which is about 10 times faster and sufficient for composite production. The matrix material was epoxy and the curing was done at 40ºC for 18 hours and 140ºC for 6 hours. The resulting composites contain 25 vol% fibres and 3 vol% porosity. The composite material was tested using a tensile testing machine. A similar composite with commercial hemp yarn was also tested. The stress-strain curves are shown opposite. The composite with fungal purified fibre bundles and the composite with the yarn have tensile strengths of 150 MPa and 170 MPa, respectively. However, the strain of the composites with fungal purified fibre bundles was 20-40% lower than the strain for the composites with yarn. Thereby stiffer composites (E-module = 24±2 GPa) can be produced with hemp fibre bundles purified with this new technique.

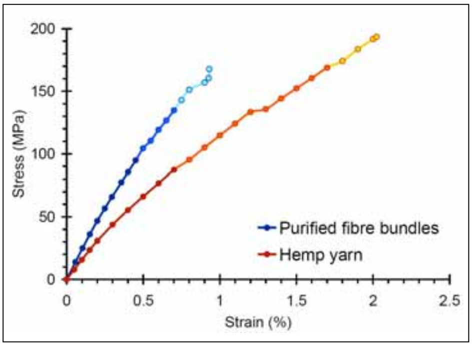
Anders Thygesen



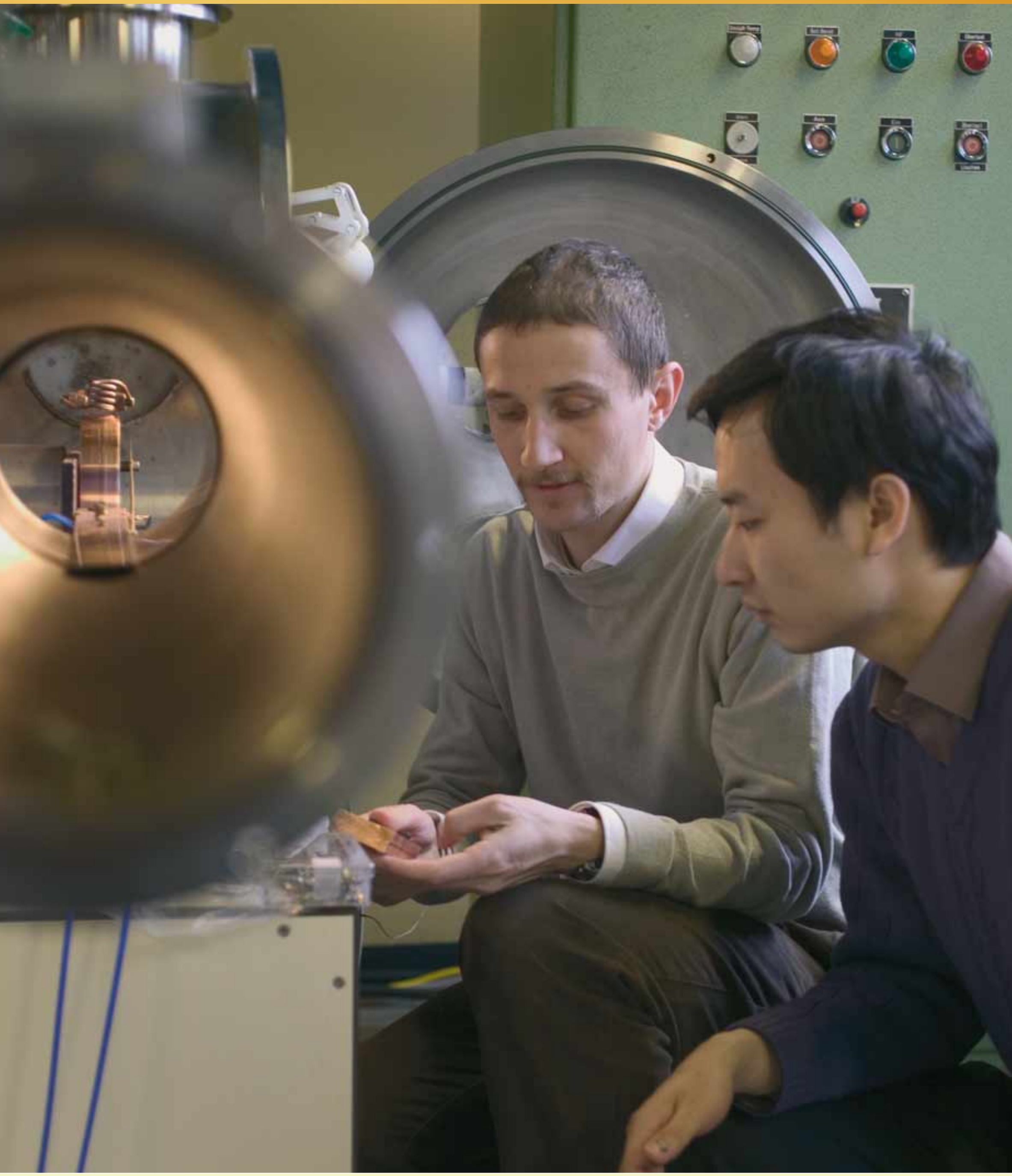
Cultivation of fungus in soil plugged with hemp stems. This fungus degrades the weak material between the fibre bundles in the hemp bast, to release fibre bundles useful for composites.



X-ray diffractogrammes for cellulose (α-cellulose), hemp bast and purified hemp fibre bundles in reflection geometry.



Stress–strain curves for composites with 25 vol% aligned hemp fibres and a matrix of epoxy. (red): Commercial hemp yarn (average of 8 curves); (blue): Fungal purified fibre bundles (average of 13 curves). The fraction of broken test specimens is indicated by the strength of the coloration: dark ~0-5%; medium ~5-49%; light ~50-95%.



Stinus Jeppesen and Ming Cheng are discussing the melt-spinning facility used for the synthesis of amorphous alloys by quenching. Recently, the set-up was redesigned to allow the production of bulk amorphous alloys by copper-mould casting. The bulk properties of specialized alloy systems are of particular interest for engineering applications and thus subject to extensive research.

Nano- and Microstructures in Materials

The research programme "Nano- and Microstructures in Materials" is concerned with work in the entire range from basic to applied research. The Programme is composed of four dominating topics: materials models and structure; radiation damage, defects and fusion materials; powder technological materials; and bulk metallic glasses. The major efforts of the mentioned topics all lie within the overall Risø focus areas "Energy" and "Industrial Technology".

Within the area of powder technological materials two important developments have been initiated and accelerated. One is work with solid-state storage of hydrogen in metal hydrides. Hydrogen is foreseen to play a major role in the future energy supply of the industrialised world. Significant initiatives have been taken by the United States of America and Japan as well as by the European Commission to promote research and development of a hydrogen economy, which may replace the present economy based on fossil fuels. The research programme Nano- and Microstructures in Materials has been active in hydrogen storage for years and these activities were intensified strongly during 2003 by involvement in several international as well as national R&D programmes. At present work is going on to identify optimal alloy systems for hydride formation with particular emphasis on development of light alloy systems, containing elements like Al, Mg and B, which may meet the generally accepted minimum requirement of 5-6 w% hydrogen content.

Another important initiative is the involvement in an innovation project aiming at development of new porous materials for use as human bone implants. The work in the project is done in collaboration between the Materials Research Department and Risø's Polymer Department as well as external partners. The project has a two-fold aim. One part of the aim, naturally, is to develop the porous materials themselves, but another aim is to merge existing, but completely different, expertises in one joint effort to establish new ideas, which may form the basis for a commercial start-up company. Currently a patent for protection of the commercial business ideas is underway and is expected to be filed during 2004.

However, the Programme includes many other important research projects and one may mention the work on materials for use in future fusion reactors. This work was presented in details in our previous annual report. Interesting findings were obtained as a result of in-reactor tensile testing of pure copper. The work has been continued during 2003 encouraged by international decisions on support to the development of fusion energy.

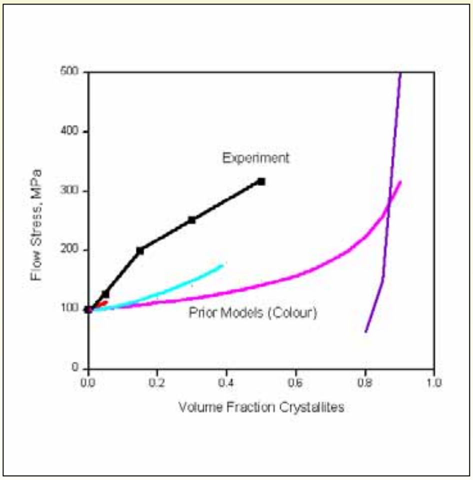
The newly commissioned JEOL 3000F transmission electron microscope has become a widely used facility for many projects in almost all programmes of the department. In addition, the microscope serves as a national centre facility accessible also for other groups in Denmark. Particularly, the University of Copenhagen, the Technical University of Denmark, and the University of Aarhus use the microscope routinely. Within this framework, the Department and the Programme are currently planning applications for new equipment to further improve the facilities of the national Danish Electron Microscopy Centre.

Allan Schrøder Pedersen

Metallic glasses

A unique property of metallic glasses compared to crystalline alloys is the dramatic softening that occurs in the temperature range just above the glass transition temperature. The ability to easily shape metallic glasses in this temperature regime is one of the most attractive characteristics of these novel materials. However, when metallic glasses are held in the temperature range where they can be readily shaped, they gradually crystallize thereby losing the ability to be shaped further.

Research examining the structure of the partially crystalline state of a metallic glass with composition $Mg_{60}Cu_{30}Y_{10}$ has been carried out in 2003. The main tool for examining the structure is the high-resolution transmission electron microscope recently installed at AFM. Microscopy revealed that nanocrystallites of the Mg_2Cu phase nucleated and gradually grew to a diameter of about 80 nm during annealing above the glass transition temperature. The partially devitrified metallic glass is thus a nanoscale composite consisting of crystallites embedded in an amorphous matrix.

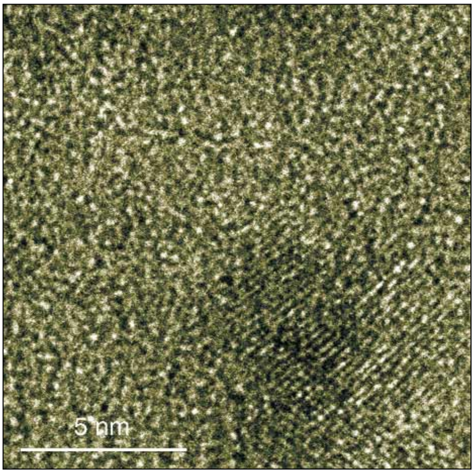


Flow stress of crystalline and amorphous nanocomposite $Mg_{60}Cu_{30}Y_{10}$ alloys in the temperature range where the material can be easily shaped. Previously proposed models for flow of emulsions and composite materials clearly show different curvatures than the experimental results and thus fail to describe our results.

An additional aspect of this work sought to compare the flow stress of the nanocomposites produced in this way with well-established models that are available for predicting the properties of composite materials. Experiments conducted in the temperature range where the material is easily shaped revealed a linear dependence of flow stress on the volume fraction of nanocrystallites. This is in contrast to predictions of a wide variety of physically based models for flow of emulsions and composite materials.

Continuation of this work seeks to develop a new physical model that accounts for the flow stress of these nanocomposite materials. In addition, exploration of the impact of crystallization on the shaping process is being explored from both technological and scientific perspectives.

John Wert

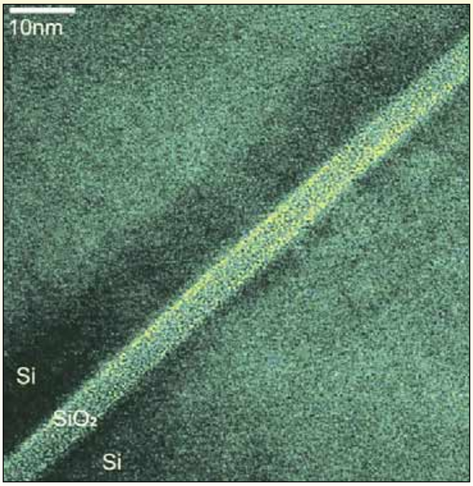


Composite of nanocrystalline and glassy $Mg_{60}Cu_{30}Y_{10}$ alloy. The lattice planes seen in the lower right hand corner identify the crystallites embedded in the amorphous matrix.

O_2 plasma assisted wafer bonding

The possibility of joining two semiconducting materials to a single, stable compound opens up a wide range of new possibilities in the production of semiconductor devices. The O_2 plasma activation technique has developed as a new method of bonding, which is particularly useful for components that contain heat sensitive materials. It is of decisive importance for future optimisation and use of this process in the industry to comprehend the mechanism behind plasma bonding.

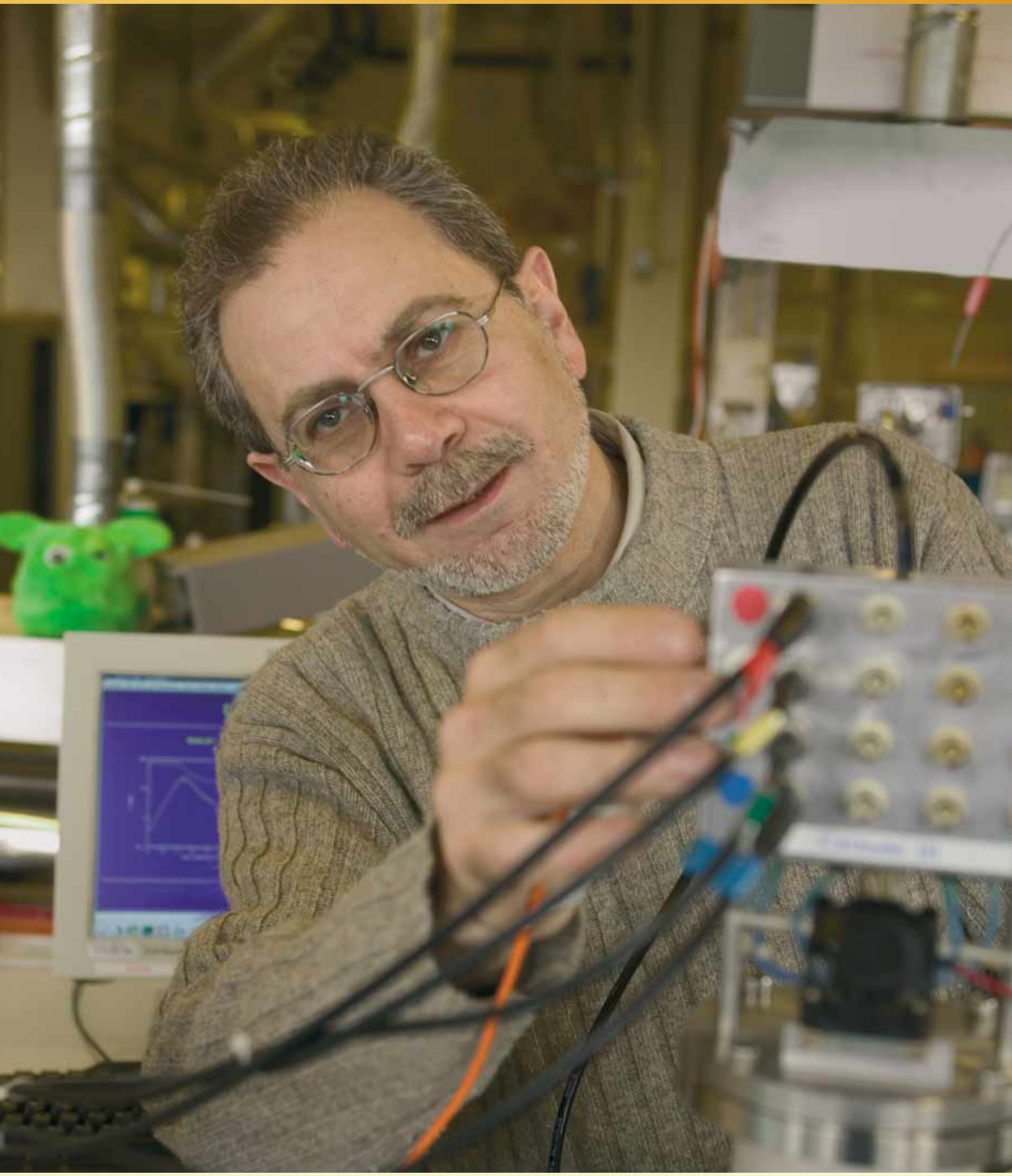
With this objective, a process has been investigated, in which two Si wafers have been plasma activated, dipped in deionised water and dried prior to bonding. The samples were prepared in a cleanroom environment at the Department of Micro- and Nanotechnology, Technical University of Denmark. The bonding occurs spontaneously, when the two wafers are adhered and the resulting bond strength is high compared to those obtained by other room temperature techniques. Hence, heating is unnecessary and the process is therefore of great interest for requests where a subsequent annealing step might hamper application. Recent X-ray diffraction experiments carried out at the Risø BW2 diffractometer at HASYLAB provide the basis of a fitted model.



A Transmission Electron Microscopy (TEM) image of an amorphous silicon oxide layer that constitutes the interface of a bonded silicon wafer pair. The limit between oxide layer and Si wafers is well defined. The thickness is uniform in all parts of the inspected sample.

To derive a detailed model of the bonding process, an algorithm that optimises the density profiles across the interface oxide was developed. In addition to the X-ray studies, the interface was examined by a number of complementary techniques: acoustic microscopy (performed in cleanroom environment at Danish Electronics, Light & Acoustics), X-ray Photoelectron Spectroscopy (XPS) and Time-of-Flight Secondary Ion Mass Spectrometry (TOF-SIMS) (in corporation with the Danish Polymer Centre) as well as Transmission Electron Microscopy (TEM). Our results show that a water treatment of the activated wafers previous to the bonding, affects only the centremost atomic layers between the two wafers. Presumably, a mono- or bi-layer remains after drying and thereby eases the bridging in plasma bonding. From TEM pictures it is evident that the oxide layer is non-porous and has well defined limits. Optimisation of the activation process and investigations of the plasma oxide have resulted in an improved and effective bonding.

Anne Egebjerg, Mette Poulsen,
Oliver Bunk, Erik Johnson and Robert Feidenhans'l



Nikos Bonanos is checking the set-up for electrochemical characterization of materials for solid oxide fuel cells, using impedance spectroscopy.

Fuel Cells and Materials Chemistry

Fuel cell systems are expected to become a key technology for energy conversion in the future. Fuel cells provide an efficient and clean conversion of chemical energy into electricity and heat. A variety of fuels can be used in solid oxide fuel cells (SOFCs), e.g. natural gas, hydrogen, bio gas, ethanol, methanol, ammonia and coal gas. Also, the fuel cell may operate in the reverse mode, i.e. as an electrolyzer (SOEC; solid oxide electrolysis cell). With an SOEC hydrogen, as well as methane and methanol, may be produced by renewable energy technologies e.g. exploiting the wind and sun.

The effort related to SOFC at Risø National Laboratory amounts to about 45 man-year/year. The work is supported by Haldor Topsøe A/S, The Danish Energy Agency through the Energy Research Programme, and The Public Service Obligation Programmes managed by the Danish electricity grid system operators, Elkraft and Eltra. Risø National Laboratory has participated in the formulation of a National Strategy for Fuel Cells, in particular for SOFCs. Risø National Laboratory is also participating in a number of European projects supported by the European Commission, e.g. in collaboration with Rolls-Royce Fuel Cell System Ltd., UK, Forschungzentrum Jülich, Germany, and Energieonderzoek Centrum Nederland.

In 2002, our pre-pilot R&D manufacturing facility was inaugurated. In 2003, the manufacture of SOFCs on a laboratory scale has been transferred successfully to our pre-pilot plant resulting in the manufacture of a large number of cells. The cells are being tested at Risø National Laboratory, Haldor Topsøe A/S, and by industries in Europe and the USA. In the laboratory, cell development has resulted in an improved cathode which may allow the operating temperature of the so-called anode-supported SOFC to be lowered to below 700ºC.

Test facilities have been expanded in 2003 such that several cell test stands can be operated for short and long term tests under various operating conditions. A close cooperation between cell testing, modelling, pre- and post-characterization and manufacture provides a speedy development. At present, the common fuel employed is methane, either pre-reformed or internally reformed.

In addition to the R&D on SOFC, the Programme is utilizing the knowledge on related energy materials and systems. This includes the already mentioned high-temperature electrolysis, oxygen membranes, gas purification (NO_x and particle removal), and magnetocaloric systems.

The education of Bachelor's, Master's and Ph.D. students as well as Post Docs is becoming an increasingly important task. The education is incorporated, primarily, in our R&D, for the benefit of all parties. During the university summer vacation, students are working in the laboratories under guidance by both young and more experienced scientists. A so-called three week course has been started in 2003 at the Technical University of Denmark, where all teaching is given by young scientists from our Programme. The students are spending about one week in our laboratories at Risø, where they experience parts of the work involved in the manufacture and testing of fuel cells. Some of the students have continued in individual courses.

In 2003, the Programme, together with five universities in Denmark, achieved a project on efficient conversion of renewable energy with the focus on SOFC and SOEC. This five year project will engage six Ph.D. students and two Post Docs. The project is expected to become an important step in the development of SOFC and SOEC.

We are proud that one of our talented Ph.D. students, Martin Søgaard, received the award 'Elektrofondets Kandidatpris' from The Society of Danish Engineers. Also in 2003, Mogens Mogensen was appointed as Research Professor at Risø National Laboratory in the area of SOFC.

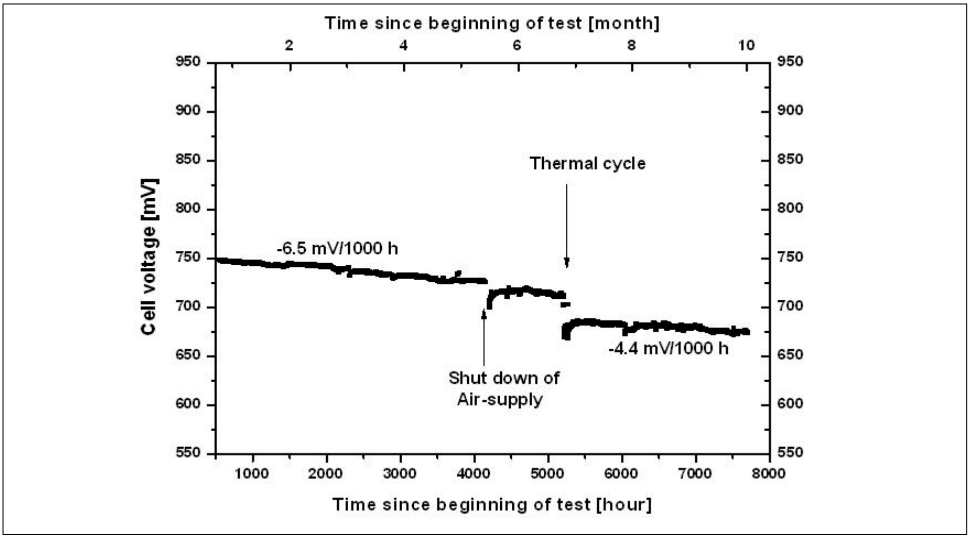
Søren Linderoth

Solid Oxide Fuel Cell testing

A focus area in SOFC development is the development of long-term stable cells and systems. The target is a system with a degradation rate of about 0.25% per 1,000 hours aiming for a total lifetime of the SOFC stack of 40,000 hours. Probably, natural gas is going to be the fuel gas applied for an SOFC-system and since direct conversion of methane is difficult, the natural gas will be reformed into an H_2-rich fuel gas through the steam-reforming process. A part of the steam reforming may take place in the SOFC anode chamber. The electrical efficiency of a fuel cell is directly proportional to the fuel utilization, and therefore an SOFC-system should operate at high fuel utilization, i.e. >90%.

During 2003, the long-term stability of the so-called second generation Risø-Topsøe SOFC has been investigated under technologically relevant conditions. The fuel is pre-reformed methane and the fuel cell is operated with high fuel utilization, above 75%. The cells were tested at various constant current densities in the interval from 0.25 A/cm^2 to 1 A/cm^2. A current density of 1 A/cm^2 is regarded as an accelerated test, while a current density of 0.25 A/cm^2 is considered mild, yet technological conditions. The rate of degradation was very dependent on the current load (or polarization). At 750ºC the rate of degradation was found to be 0.25 %/1,000 hours at a current density of 0.25 A/cm^2 and as high as 12 %/1,000 hours at 0.75 A/cm^2. The mechanism for the degradation is not understood fully, but possible mechanisms like Ni-coarsening and Ni loss at the anode may account for a part of the degradation. More investigations are necessary before a model for the degradation can be made. To demonstrate real long term operation, a single cell has been tested under constant conditions for >6,000 hours. The cell is tested at 850ºC at a constant current density of 1 A/cm^2. The rate of degradation is found to be <1% per 1,000 hours. After 5,000 hours of test the cell experienced a mild temperature cycle which resulted in a drop in the performance. This is ascribed to the test set-up rather than the cell, and a future task for the test group is to develop test facilities that allow thermal cycling without degradation. The change in performance over time for the 6,000 hours test is illustrated in the figure.

Rasmus Barfod



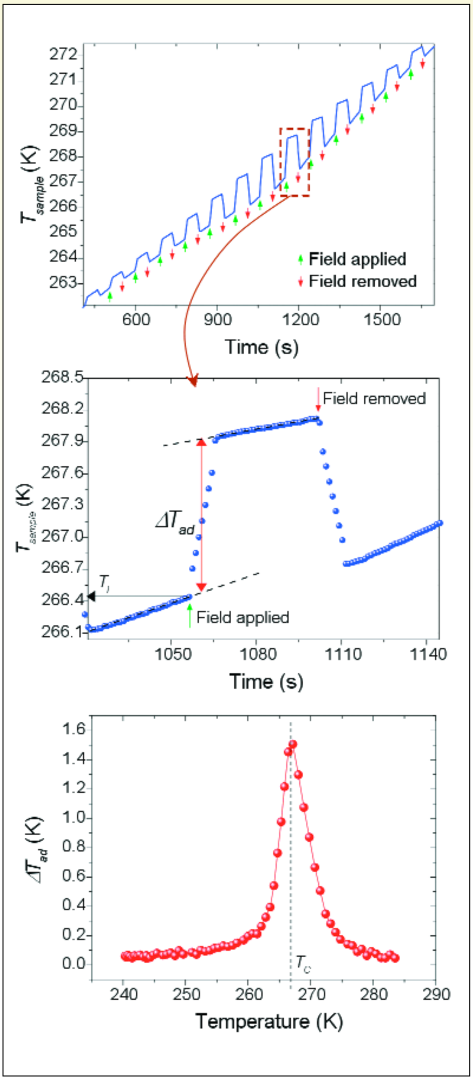
The change in performance over time of test for a cell tested for approximately 10 months.

Magnetocaloric properties of $La_{0.67}Ca_{0.33-x}Sr_xMnO_{3\pm\delta}$

Magnetic cooling near room temperature is an upcoming technology, which may prove to be both more efficient and more environmentally friendly than conventional gas cooling. A key challenge is to develop magnetic refrigerants, which apart from showing a large magnetocaloric effect around room temperature are chemically stable, non-toxic, and not too expensive. The present study regarded the magnetocaloric properties of $La_{0.67}Ca_{0.33-x}Sr_xMnO_{3\pm\delta}$ ($x \in [0;0.33]$). The magnetocaloric effect or, more precisely, the adiabatic temperature change ΔT_{ad} due to a change of the external magnetic field, was measured by moving the sample in and out of a magnetic field ($\mu_0 H = 0.7$ T) continuously monitoring the sample temperature.

The sample was placed in a Teflon® container to ensure near-adiabatic conditions and a cryostat system was used to control the initial sample temperature. The figure shows results obtained for the $La_{0.67}Ca_{0.33}MnO_{3\pm\delta}$ compound, which exhibits a large magnetocaloric effect near the Curie point (T_C = 267 K), where its magnetic spin system is most susceptible to changes of the external field. In fact, the effect is larger than the magnetocaloric effect observed for the prototypical material for room temperature magnetic refrigeration, the metal gadolinium (Gd). Unfortunately, ΔT_{ad} is vanishing at room temperature. It was found, however, that the Curie temperature, and thereby the magnetocaloric temperature working range, could be increased by making a partial substitution of Ca with Sr. Varying the Sr/Ca ratio, any T_C value between 267 and 400 K could be obtained. Together, the significant magnetocaloric effect, the possibility of adjusting the temperature working range, and the excellent chemical stability of $La_{0.67}Ca_{0.33-x}Sr_xMnO_{3\pm\delta}$ make these compounds suitable candidates as refrigerants in future magnetic cooling devices operating near room temperature.

Anders Reves Dinesen



The magnetocaloric effect measured for $La_{0.67}Ca_{0.33}MnO_{3\pm\delta}$. Top figure: Temperature profile recorded during a sequence of magnetization cycles performed while slowly increasing the sample temperature. Middle figure: Enlarged view of a single magnetization cycle. Bottom figure: The measured adiabatic temperature change as a function of temperature.

24th Risø International Symposium on Materials Science

A group of 100 participants from 16 different countries came to Risø National Laboratory for the symposium “Superconductivity and Magnetism: Materials Properties and Developments” on the 10th to the 13th of September. The participants constituted an interesting mix of students, young researchers and senior scientists. As many as 24 invited prominent scientists accepted the invitation and contributed to an atmosphere of enthusiasm and discussion.

One of the hot topics of the conference was the strongly debated mechanism underlying high-T_c superconductivity. Experimental and theoretical presentations covered this topic from different angles. Another topic was the superconducting compound, MgB_2, which was discovered two years ago. It is a promising candidate for the next generation of commercial superconductors, and various aspects of this field were discussed. In addition, there were several interesting presentations on basic and applied science within giant magneto-resistance materials, spintronics, nanomagnetism, and biophysics.

The social activities of the symposium were good opportunities for students and scientists to establish and renew personal contacts. The symposium dinner was held on board the old steamer "Sagafjord" cruising Roskilde Fjord. This was a perfect setting for the many participants to get to know each other and discuss more physics. Furthermore, the Danish history was studied at the Viking Ship Museum.

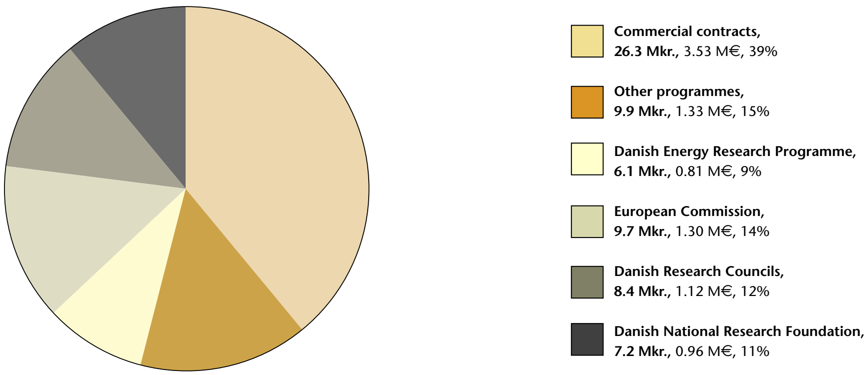
Katrine Nørgaard Toft

The Symposium inspired intense scientific discussions extending through a beautiful boat trip on Roskilde Fjord.

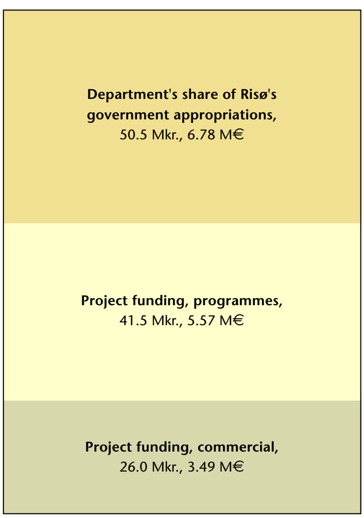
Finances

The activities of the Department are supported by a combination of government funding, focused project funds from national and international research programmes, and fully commercial contracts.

Project Income

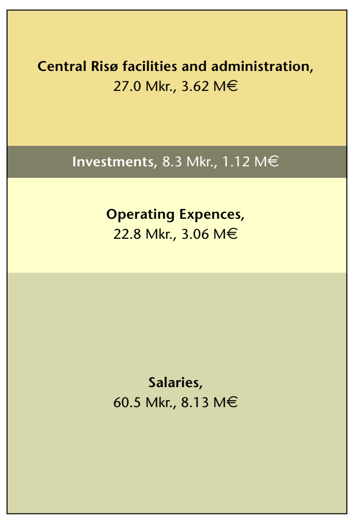


Income



Total: 118.0 Mkr., 15.8 M€

Expenditures



Total: 118.6 Mkr., 15.9 M€

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Shigekazu, Morito
Kashihara, Keizo
Xingpin, Chen
Xu, Min

Emeritus
Hansen, Niels
Leffers, Torben*
Nielsen, Mourits

Persons leaving (#) or joining (*) the Department during 2003. (§) Employed at the University of Copenhagen.

Educational work

The Department is strongly involved in the education of students at different levels. The involvement ranges from postgraduate and undergraduate courses and projects, in collaboration with universities and industry, to lectures for high school classes. A summer student programme was organized and a number of study groups on various subjects are taking place continuously within the Department. Several international workshops and summer schools aimed at Ph.D. students have been arranged. In addition, many staff members of the Department act as external university lecturers and examiners. The education of Ph.D. students and that of Master's and Bachelor's Degree students are some of the most important educational activities. During 2003 six Ph.D. students completed their Ph.D. projects, while 21 projects were still ongoing, in addition to 32 Master's or Bachelor's Degree students.

Katrine Nørgaard Toft near the end.

Ph.D. projects completed in 2003

Asger Abrahamsen
"Possible magnetism in vortex cores of super conducting $TmNi_2B_2C$ studied by small angle neutron scattering". Technical University of Denmark. Supervisors: Claus Schelde Jacobsen (DTU), Jørn Bindslev Hansen (DTU), Niels Hessel Andersen.

Jesper Christiansen
"Dislocation interactions with surfaces and grain boundaries". Technical University of Denmark. Supervisors: Karsten Wedel Jacobsen (DTU), Jakob Schiøtz, Torben Leffers.

Anders Reves Dinesen
"Magneto-caloric and magneto-resistive properties of $La_{0.67}Ca_{0.33-x}Sr_xMnO_3$". Technical University of Denmark. Supervisors: Steen Mørup (DTU), Søren Linderoth, Nini H. Pryds.

Christian Højerslev
"The influence of microstructure on the fatigue properties of high strength materials for cold forging tools". Technical University of Denmark. Supervisors: Marcel Somers (DTU), Povl Brøndsted, Jesper Vejlø Carstensen.

Lars Mikkelsen
"Oxidation of iron-chromium alloys". University of Southern Denmark. Supervisors: Eivind Skou (SDU), Søren Linderoth, Mogens Mogensen, Peter Halvor Larsen.

Trine Bjerre Pedersen
"Modelling of residual stresses in sprayformed structures". Technical University of Denmark. Supervisors: Jesper Hattel (DTU), Nini H. Pryds.

Ongoing Ph.D. projects

Anders Andreasen
"Preparation and characterization of new metal hydrides for hydrogen storage". Technical University of Denmark. Supervisors: Ib Chorkendorff, (DTU), Søren Dahl (Haldor Topsøe A/S), Flemming Besenbacher (AU), Allan Schrøder Pedersen.

Jens W. Andreasen
"Nano-structured catalysts". Technical University of Denmark. Supervisors: Kenny Ståhl (DTU), Alfons Molenbrock (Haldor Topsøe A/S), Robert Feidenhans´l.

Christian Robert Haffenden Bahl
"Neutron, X-ray and TEM studies of magnetic nano-particles". Technical University of Denmark. Supervisors: Steen M Mørup (DTU), Kim Lefmann and Luise Theil Kuhn.

Niels Bech Christensen
"Quantum phase transitions". University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Des McMorrow.

Carsten Gundlach
"Recovery in aluminium". University of Copenhagen. Supervisors: Erik Johnson (KU), Henning Friis Poulsen, Wolfgang Pantleon.

Anette Nørgaard Hansson
"Protection against metallic oxidation". Technical University of Denmark. Supervisors: Marcel Somers (DTU), Søren Linderoth, Mogens Mogensen.

Jens V. T. Høgh
"Kinetics of metal/hydrogen electrode on solid electrolytes". Technical University of Denmark. Supervisors: Torben Jacobsen (DTU), Ib Chorkendorff (DTU), Mogens Mogensen.

Søren Højgaard Jensen
"High temperature electrolysis". Technical University of Denmark. Supervisors: Ib Chorkendorff (DTU), Mogens Mogensen, Peter V. Hendriksen, Nikolaos Bonanos.

Thomas Bagger Stibius Jensen
"Neutron and Synchrotron X-ray studies of charge and magnetic ordering in superconductors". University of Copenhagen. Supervisors: Per Hedegaard (KU) and Niels Hessel Andersen.

Stine Nyborg Klausen
"Magnetic dynamics of nanoparticles". Technical University of Denmark. Supervisors: Steen Mørup (DTU), Kim Lefmann, Kurt N. Clausen.

Trine Klemensø
"Relationships between structures and performance of SOFC anodes". Technical University of Denmark. Supervisors: Torben Jacobsen (DTU), Jørgen Gutzon Larsen (Haldor Topsøe A/S), Mogens Mogensen.

Tine Knudsen
"Hot Deformation Structures in Metals". Roskilde University Centre. Supervisors: Jeppe Dyre, Grethe Winter, Niels Hansen.

Axel W. Larsen
"Nucleation in metals". Technical University of Denmark. Su pervisors: Jens Als-Nielsen (KU), Henning Friis Poulsen, Dorte Juul Jensen.

Zhengjie Li
"Effect of Grain Orientation on Dislocation Structures and Mechanical Properties". Technical University of Denmark. Supervisors: Niels Bay (DTU), Grethe Winther, Niels Hansen.

Bo Madsen
"Properties of plant fibre composites – an experimental model study". Supervisors: Lars Damkilde (DTU), Preben Hoffmeyer (DTU), Anne Belinda Thomsen (PRD, Risø), Hans Lilholt.

Mette Poulsen
"Nanostructuring with bonding". Technical University of Denmark. Supervisors: Flemming Jensen (DTU), Robert Feidenhans´l.

Henrik Nikolaj Blicher Schmidt
"Modelling of mechanical and metallurgical properties of friction stir welded joints". Technical University of Denmark. Supervisors: Jesper Hattel (DTU), John A. Wert.

Martin Søgaard
"Properties of perovskites with varying A/B-ratio". Technical University of Denmark. Supervisors: Torben Jacobsen (DTU) , Peter Vang Hendriksen, Mogens Mogensen, Finn Willy Poulsen.

Anders Thygesen
"Hemp fibres for light and strong composites – optimisation and characterisation". Royal Veterinary and Agricultural University. Supervisors: Per Ole Olesen (KVL), Claus Felby (KVL), Anne Belinda Thomsen (PRD, Risø), Hans Lilholt.

Katrine Nørgaard Toft
"Magnetic properties of superconductors". University of Copenhagen. Supervisors: J. Jensen (KU), Niels Hessel Andersen.

Christian Bech Wejdemann
"Nanomechanics of metal fatigue". Technical University of Denmark. Supervisors: Karsten W. Jacobsen (DTU), Ole Bøcker Pedersen, J. B. Bilde-Sørensen.

Master's or Bachelor's projects

Peter Andersen
"Antiferromagnetism in YBCO nanoparticles". University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Luise Theil Kuhn.

Jens Blaabjerg, Kaare Kaaber
"Pattern recognition algorithms for the 3DXRD microscope". University of Copenhagen. Supervisors: Peter Johansen (KU), Søren Schmidt.

Anja Buhrkall
"Use of cast iron in windmills". Engineering School Copenhagen. Supervisors: Gitte Mellemgaard (IHK), Krin Sigumfeldt (IHK), Jacob Ilsted Bech, Povl Brønsted.

Bitten Plesner Christensen, Jacob Kirkensgaard Hansen, Kåre Stokvad Hansen
"Optimering af neutronspredningsudstyr ved Monte Carlo simulering". Roskilde University. Supervisors: Dorthe Posselt (RUC), Kim Lefmann.

Sophie Decourcelle
"pH controlled dispersion of Al_2O_3 in ethanol", ENSCI, Limoges, France. Supervisors: Mohan Menon, Séverine Ramousse, Peter H. Larsen. Completed in 2003.

Henrik Edwards
"Monte Carlo simulation of neutron scattering from high T_c superconductors". University of Copenhagen. Supervisors: Jens Als-Nielsen (KU), Kim Lefmann.

Anne Egebjerg
"X-ray scattering investigations of bonded interfaces". University of Copenhagen. Supervisors: Sine Larsen (KU), Flemming Jensen (DTU), Robert Feidenhans'l.

Lars Anders Fledelius, Henrik Tomra Skovsgaard Hegner Jensen
"Development of TACO device servers for data processing in FABLE". Engineering College of Copenhagen. Supervisors: Søren Schmidt, Henning Haugaard (Engineering College of Copenhagen). Completed in 2003.

Ditte Gundermann, Tobias Larsen, Martin Graversgaard Nielsen, Neslihan Saglanmak
"Synthesis of Fe-Au nano particles by the micelle method". Roskilde University. Supervisors: Niels Boye Olsen (RUC), Luise Theil Kuhn.

Britt Rosendahl Hansen
"Properties of magnetic nano-particles". Technical University of Denmark. Supervisors: Steen Mørup (DTU) and Kim Lefmann.

Karen-Anne Herdahl, Heidi Kolmorgen Nielsen
"Exact numerical diagonalisation of magnetic Bloch oscillations in one dimension". Roskilde University. Supervisors: Jeppe Dyre (RUC), Kim Lefmann. Completed in 2003.

Jari í Hjøllum
"Maghemite and hematite nanoparticles, synthesis and magnetic properties". University of Copenhagen. Supervisors: Morten Bo Madsen (KU), Luise Theil Kuhn.

Joris Hofhuis
"Synthesis and characterisation of metal hydrides and metal-oxy-hydrides". Delft University of Technology. Supervisor: Finn W. Poulsen. Completed in 2003.

Annemarie Huijser
"Isotope effect in proton conducting oxides". Delft University of Technology. Supervisors: Finn W. Poulsen, Nikolaos Bonanos. Completed in 2003.

Birgitte Abery Jacobsen
"Critical currents in BiSCCO/Ag tapes for superconducting

power cables”. University of Copenhagen. Supervisors: P.E. Lindelof (KU), Jørn Bindslev Hansen (DTU), Niels Hessel Andersen.

Søren Højgaard Jensen
“High temperature solid oxide electrolyser”. University of Copenhagen. Supervisors: Jan W. Thomsen (KU), Mogens Mogensen. Completed in 2003.

Stinus Jeppesen
“Structural and magnetic properties of amorphous alloys”. Technical University of Denmark. Supervisors: Steen Mørup (DTU), Nini Pryds

Lasse Jonsen
”Crack growth in gel-coat”. Eng. School Odense Teknikum. Supervisors: Mariann Niss (IOT), Povl Brøndsted.

Kenneth Jørgensen
“Modelling of delamination at ply drops”. Technical University of Denmark. Supervisors: Viggo Tvergaard (DTU), Bent Sørensen.

Thomas Jørgensen
“Investigation of the superconducting properties of MgB_2/Fe tapes”. University of Copenhagen. Supervisors: Per Hedegård (KU), Jean-Claude Grivel. Completed in 2003.

Frantz Bræstrup Radzik
“Development and studies of ladder compound super conductors”. University of Copenhagen. Supervisors: Emil Makovicky (KU), Jean-Claude Grivel and Niels Hessel Andersen.

Mikkel Salling
“Mapping of in-plane conductivity fuel cell current collectors”. Technical University of Denmark. Supervisors: Gøsta Tuesen (DTU), Uffe Korsbech (DTU), Nikolaos Bonanos.

Casper Thorning
”Orientation subdivision of grains of known orientation in polycrystals strained in tension”. Technical University of Denmark. Supervisors: Marcel A.J. Somers (DTU), John Wert.

Rasmus Torekov
“Development of a test rig for composite slab under compressive loads”. Technical University of Denmark. Supervisors: Peder Klit (DTU) and Jacob Ilsted Bech.

Lecturing courses and other teaching activities

Basic course in fuel cell
DTU, course no. 41410, 2003. Lecturers: D. Lybye, S. Ramousse, R. Barfod.

Composit Materials
Roskilde Technical College/HTX. Lecturer: Lars P. Mikkelsen.

Composit Materials and Processing
Odense University College of eng., autumn 2003. Lecturers: Lars P. Mikkelsen and Bent F. Sørensen.

Current topics in Physical Metallurgy
Risø, spring 2003. Lecturer: Roger Doherty.

DC-Characteristics of SOFC
DTU Fagpakkeprojekt. May 2003. Advisor: Rasmus Barfod.

Defect Chemistry Course
Universität Karlsruhre. Nov. 2003. Lecturer: Finn W. Poulsen.

Energy – Fuel Cells and the Hydrogen Society
DTU, course no. 41403, 2003. Lecturer: Mogens Mogensen.

Engineering Physics - Solid Oxide Fuel Cells
Technical University of Helsinki. Nov. 2003. Lecturer: Finn W. Poulsen.

Mathematics A, Excersises
KU. Autumn 2003. Lecturer: Axel W. Larsen.

Modern Physics
DTU, course no. 10501, 2003. Lecturer: Peter V. Hendriksen.

Nano Magnetism
Study group for Ph.D. students and others, Risø, DTU and NBI, 2003 Lecturers: Stine N. Klausen, K. Lefmann, Per-Anker Lindgård, Luise Theil Kuhn.

Nano Science Seminars
for Ph.D. students and others, NBI, 2003. Member of organising committee: Luise Theil Kuhn.

Positron Annihilation CNRS School
CERI-CNRS, Orle’ans, France Nov. 20-21, 2003. Lecturer Morten Eldrup.

Physical Metallurgy
DTU, course no. 42150, 2003. Lecturer: John Wert.

QUP-Seminars
DTU, 2003. Lecturer: Per-Anker Lindgård.

SOFC-brændselsceller
Nørremarksskolen, Vejle. 2003. Lecturer: Dorthe Lybye.

Solid oxide fuel cells
DTU, 2003. Lecturer: Séverine Ramousse.

Structure and Solid State Chemistry
DTU, course no. 26320, 2003. Lecturer: Finn W. Poulsen.

Superconductivity
for masters students at Copenhagen University, autumn 2003, Experimental supervisors: Jean Claude Griviel, Bente Lebech, Luise Theil Kuhn, Asger Bech Abrahamsen, Kim Lefmann and Niels Hessel Andersen; theoretical supervisor: Thomas Bagger Stibius Jensen and Per Hedegaard (KU).

Tillämpad Kärnfysik
Lunds Universitet, Sweden, autumn 2003.
Lecturer: Kim Lefmann.

X-ray Physics
Course for Ph.D. and master students, NBI, 2003. External Lecturer: Desmond F. McMorrow, excercises: Martin Meedom Nielsen and Oliver Bunk.

Organization of international meetings

International Workshop on Hard Synchrotron X-ray for Texture and Strain Analysis
DESY, Germany, Apr. 9-11, 2003. Member of Committee: Jensen, D.J. Chairman: Poulsen, H.F.

International Conference on the Strength of Materials
Budapest, Hungary, Aug. 25-30, 2003. International Committee: Jensen, D.J.

24th Risø International Symposium Superconductivity and magnetism: Materials properties and developments
Risø National Laboratory, Denmark, Sep 10-13, 2003. Organizers: Andersen, N.H.; Lebech, B.; Larsen, A.; Bay, N.; Grivel, J.-C.; Hedegård, P.; McMorrow, D.; Mørup, S.; Kuhn, L.T.; Lefmann, K.; Lindelof, P.-E.; Linderoth, S.; Pedersen, N.F.

11th International Conference on Fusion Reactor Materials
Kyoto, Japan, Dec. 7-12, 2003, Member of program Committee: Singh, B.N.

Prizes, awards, honours

Per-Anker Lindgård
Appointed Adjunct Professor, Department of Physics, Danish Technical University, Lyngby

Desmond F. McMorrow
Received the Allan Mackintosh Award

Mogens Mogensen
Appointed Research Professor at Risø National Laboratory

Martin Søgaard
Received the "Elektrofondets Kandidatpris" from the Society of Danish Engineers

Teaching in the Department

Many scientists in the Department are involved in teaching activities - beyond that of supervising Ph.D. and Master's degree students. The experimental facilities and the expertise are hereby shared with many potential professionals. There are courses for undergraduates, for high school students and for summer students. The latter is an excellent way for young people to get acquainted with the Risø spirit. To give an impression, here are some examples from the various groups:

In the autumn term, two senior scientists shared the pleasure of going to Odense by train every Monday teaching *Composite materials and processing* for students studying for B.Eng. in Integrated Design. The course is a mixture of theory, experiments (done at Risø) as well as visits to industrial companies (LM Glasfiber A/S and Fiberline Composites A/S). While the students sometimes were a bit lost in the mathematics they also expressed that the ”nerds” from Risø were a welcome change in their study. More than 30 students attended the course in 2003.

A course on *superconductivity* was given at Copenhagen University with a broad course on relevant experimental techniques at Risø, and some part at DTU. Eight staff members participated extensively at various stages of the course. The nine students were very enthusiastic and obtained excellent results at the following examination. A special challenge was offered: Who makes the best superconductor? and the winning team was awarded a prize. Three of the students consider continuing with their Master's thesis work on superconductivity at Risø.

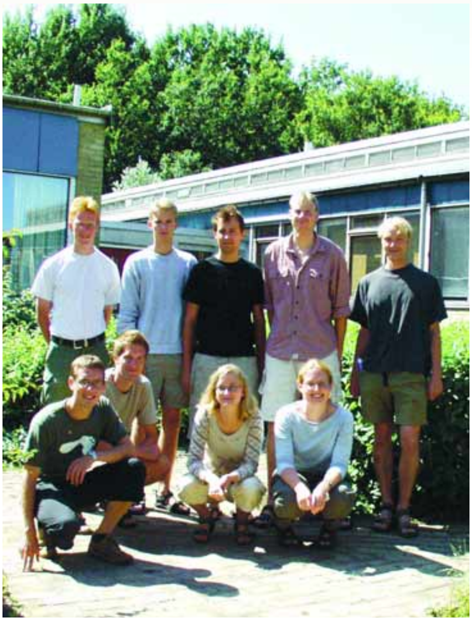
For three of the scientific staff in the fuel cell group, year 2003 started with teaching at a three weeks course at DTU: *Basic course on fuel cells*. The course is a combination of lectures at DTU and experimental work at Risø. The students were very positive and expressed that they were pleasantly surprised to meet the young and enthusiastic teachers who could pass on their knowledge in a very down to earth way. Six of the 18 students continued at Risø with smaller projects afterwards, and two as summer students. High school classes have also visited the group and a ninth grade physics class had an introduction to SOFC.

A full semester course was given by a visiting professor at the M4D centre at Risø on *Current Topics of Physical Metallurgy.* Eight students attended the course and utilised the possibility of gaining the university-approved credit.

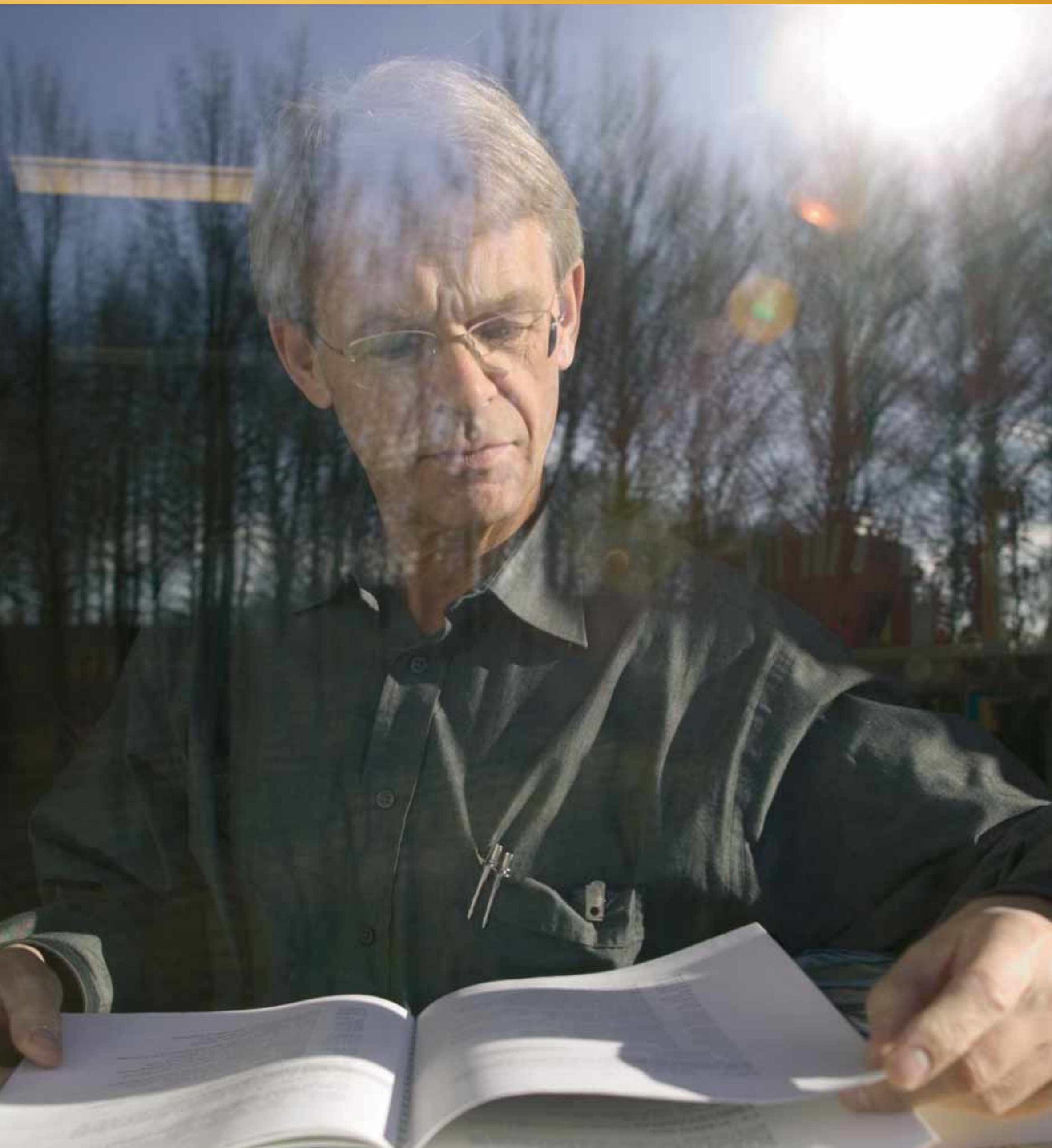
The Summer Student Program

A number of projects for undergraduates have been offered, which enables them to work closely on an active research project and to get a feel for what research is about. Usually, it amounts to six to eight weeks during the summer, hence the name, but might also be at other times. About 30 undergraduate students from University of Copenhagen, Technical University of Denmark and Roskilde University and elsewhere participated. Most students terminated their projects with a written report and a seminar. Several of the students learned to use a scanning electron microscope for their projects. As one expressed his appreciation: “It is cool to learn how to use such a microscope”.

Per-Anker Lindgård



Some of the summer students.



Morten Eldrup takes a critical look at the manual for the PC programme package PATFIT-88, which has been developed at Risø for the analysis of positron annihilation spectroscopy data. Until now, the programme package has been distributed, on a commercial basis, to more than one hundred research groups around the world.

Published work

International Publications

Algers, J.; Maurer, F.H.J.; *Eldrup, M.;* Wang, J.S., Free volume and mechanical properties of Palacos (R) R bone cement. *J. Mater. Sci. - Mater. Med.* (2003) v. 14 p. 955-960

Alonso, C.; Gurney, R.W.; Eliash, R.; Hong, S.C.; Shen, Y.R.; Jensen, T.R.; *Kjær, K.;* Konovalov, O.; Lahav, M.; Leiserowitz, L., A crystalline Langmuir monolayer designed as a template for selective intercalation of water soluble a-amino acids. *Cryst. Growth Des.* (2003) v. 3 p. 683-690

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Bilde-Sørensen, J.B.; Schiøtz, J., Nanocrystals get twins. *Science* (2003) v. 300 p. 1244-1245

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Christiansen, G.; Bowen, J.R.; Lindbo, J., Electrolytic preparation of metallic thin foils with large electron-transparent regions. *Mater. Charact.* (2002) v. 49 p. 331-335

Clausen, B.; *Leffers, T.; Lorentzen, T.*, On the proper selection of reflections for the measurement of bulk residual stresses by diffraction methods. *Acta Mater*. (2003) v. 51, 6181-6188

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Kawano, S.; Takahashi, M.; Shigeoka, T.; Iwata, N.; Shiimoto, M.; *Lebech, B.,* Two-dimensional magnetic structures and the multistep metamagnetic phases in $TbRu_2Si_2$, p. 51-52

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